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14. ABSTRACT
The objectives include depositing a thin, dense glass layer onto a porous ceramic substrate which is permeable to hydrogen only. Clay substrates (approx. 35 mil thick), suitable for glass coating, were successfully prepared and sintered. A dense glass coating, applied to the clay using an air brush, showed no permeability to air at room temperature. Comparative uncoated clay samples showed permeability. Pre- and post sintering were performed in a tube furnace without producing cracks. In addition, electrophoretic deposition of silica onto clay discs was performed in an attempt to accurately control the layer thickness. High temperature test apparatus was assembled.

15. SUBJECT TERMS
glaze, kaolin clay, tube furnace, permeance, electrophoretic deposition, rice starch, grog

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CONTRACT C100 OPTION FINAL REPORT

NAVY STTR N04-T034

1 OBJECTIVE

Our objective for the Phase I option is to deposit a thin, dense glass layer onto a porous ceramic substrate which is permeable to hydrogen only.

2 APPROACH

Our original approach to obtaining a suitable porous substrate for glass deposition has changed. Due to inconsistencies with the Cotronics ceramic casting compound we developed clay substrates and worked toward improving their permeability. In addition, we pursued alternatives for glass deposition including glaze application and electrophorectic deposition. Plasma enhanced chemical vapor deposition results were not yet complete.

3 ACCOMPLISHMENTS

- Achieved dense glass coating on clay substrate with one spray application of fritted glaze using an air brush. Comparative uncoated sample showed permeability.
- Performed electrophoretic deposition of silica onto clay sample discs.
- Successfully prepared clay substrates (approx. 35 mil thick) suitable for glass coating
- Successfully sintered coated substrates without cracks or pinholes.

4 CONCLUSION

- Cotronics formula 940LE cannot be made consistently hole free.
- Clay can be made porous using organic starches such as rice flour.
- The permeance is currently within the same order of magnitude of commercially available materials.
- IBAD processing affects the surface of the ceramic requiring pre-sintering.
- Glaze was successfully applied to a clay substrate obtaining a dense coating.
- We have not done high temperature testing.
- Test rig is build is being completed.
- EPD can be used to deposit glass but surface energy is important.

- Glass flows during sintering which is advantageous for depositing into pores.
- Soak or dwell time may be important to prevent too much run off.

5 WORK PERFORMED

5.1 Intermediate layer development

The following section describes the work performed to date on ceramic support layers. The target properties for the support layer are a 5 micron pore size, less than 1 psi pressure drop, capability to withstand reformer temperatures of ~1,800 deg F and a matching coefficient of thermal expansion to glass. Development work includes cast layers of Cotronics 940LE, roll formed kaolin clay and Ceramatec C-16 alumina.

5.1.1 Cast samples

Previous phase I work on casting highlighted the difficulties of obtaining hole free surfaces. The Cotronics 940LE high temperature ceramic adhesive is aqueous based and generates bubbles from a chemical reaction when the activator is added to the dry powder. Methods employed to dissipate the bubbles included mechanical vibration, ultrasonic agitation in a liquid bath, chemical bubble breakers and evacuation in a bell jar using a vacuum pump. All methods were inconsistent. It was believed that the particle interaction in the mixture played a significant role in the resulting surface properties. The mixing of the liquid into the dry powder, along with the liquid/powder ratio affected the viscosity of the compound. This in turn led to cracks if the mixture was too wet and pinholes or unburst bubbles if the mixture was too dry. A separate issue of surface tension leading to raised craters (a common problem in glazes) was resolved by the addition of surfactants. These results were very consistent.

While working with clays we found in the literature that starches, such as wheat, corn or rice flour, can absorb liquid faster than some of the other dry powders. Therefore, they can significantly affect the viscosity of the mixture depending on the order the ingredients are added. In clays the starch is added after the clay and water are mixed otherwise the starch would absorb too much water and swell leaving little water for the clay. In our work with the 940LE we were adding the starch after the activator, water and soap. The starch could be absorbing components in the activator. When we varied the order of mixing so the starch and water were mixed prior to the activator, the viscosity changed to a smoother consistency. When the activator is added last to the liquids, the starch grains seem to agglomerate so it’s clear that the activator is affecting the particle interaction.

To improve the mixing step and obtain more consistent results, acid (pH 4 methane sulfonic) was added as a dispersant to improve the colloidal properties. The acid (4% by weight) is added as the final liquid component. Then the ceramic powder is mixed in slowly. While the acid reduced the number of holes in the surface, it did not yield perfect results. The surface was smooth in the center but rough in the outer region. When comparing cast coupons to castings with a monolith, the monolith samples had a smoother surface. Since the monoliths are pre-moistened, the drying time is lengthened. This led us to experiment with increased drying time. The coupons were placed in a cool, humid environment. While the results showed improvement, no crusty region at the perimeter, there were still the occasional holes. Increasing acid content only led to cracking.

5.1.1.1 Cast samples permeation test results

Table 1 Flow vs. pressure data for S/N 0031 cast 940LE disc

Ptot Psig	DELP Psi	SCCM
0.64	0.62	962
1.31	0.47	870
2.27	0.36	735
3.3	0.25	587
4.13	0.17	404
4.7	0.1	235
5.48	0.02	18.69
7.55	0.11	337
10.2	0.13	436

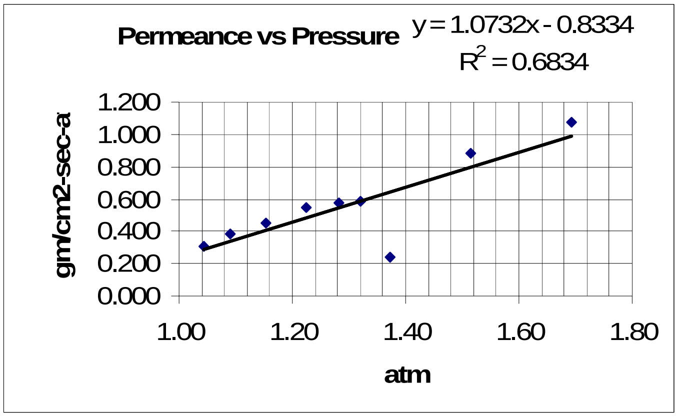

Figure 1 S/N 0031 Permeance vs. Pressure

The data in Table 1and Figure 1 for S/N 0031 shows the high flow rates achieved with the Cast 940LE samples. The flow is so high that the differential pressure never climbs above 1 psi. This is an indication that there are large holes in the surface.

5.1.2 Clay samples

Clay was pursued as a means to provide a ceramic substrate with small pores that was free from surface holes. A brief study was made of the weight loss of clay test discs from

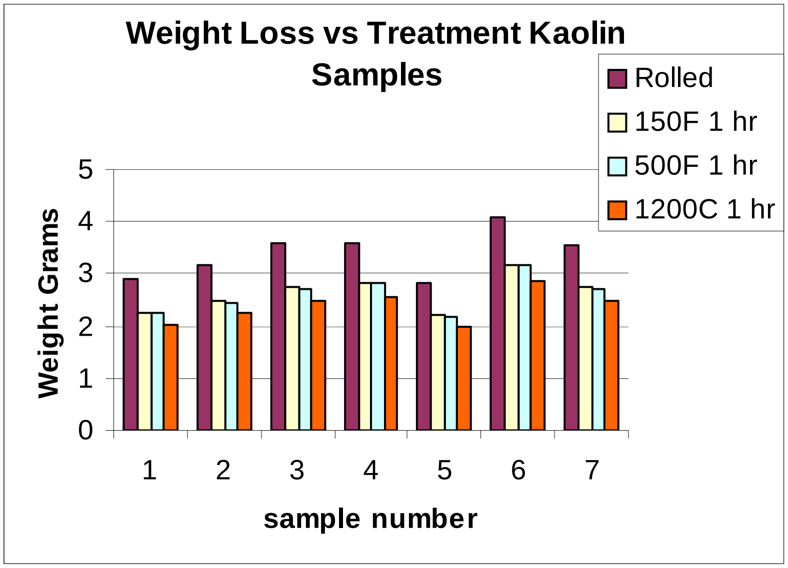

Figure 2 Clay disc weight variation with heat treatment

the as rolled condition to the 150F dried, 500F dried and 1200C fired conditions. Seven sample discs were prepared and their weights were recorded after each of the treatments. The results are shown in Figure 2. The average weight at the end of the drying step 260 C (500F) for 2 hours is 77.3% and after firing is 70.3% and the standard deviation shows the correlation is excellent

5.1.2.1 Forming & Drying

The mixing was accomplished by simply working the mixture by hand. The sample was rolled between sheets of saran (polybutadiene) film to a thickness of ~40 mil. After the discs were punched out, the supporting film was removed. The as rolled samples were dried at 150 F for about 2 hours and 500 F for about 2 hours. Several Photomicrographs were taken.

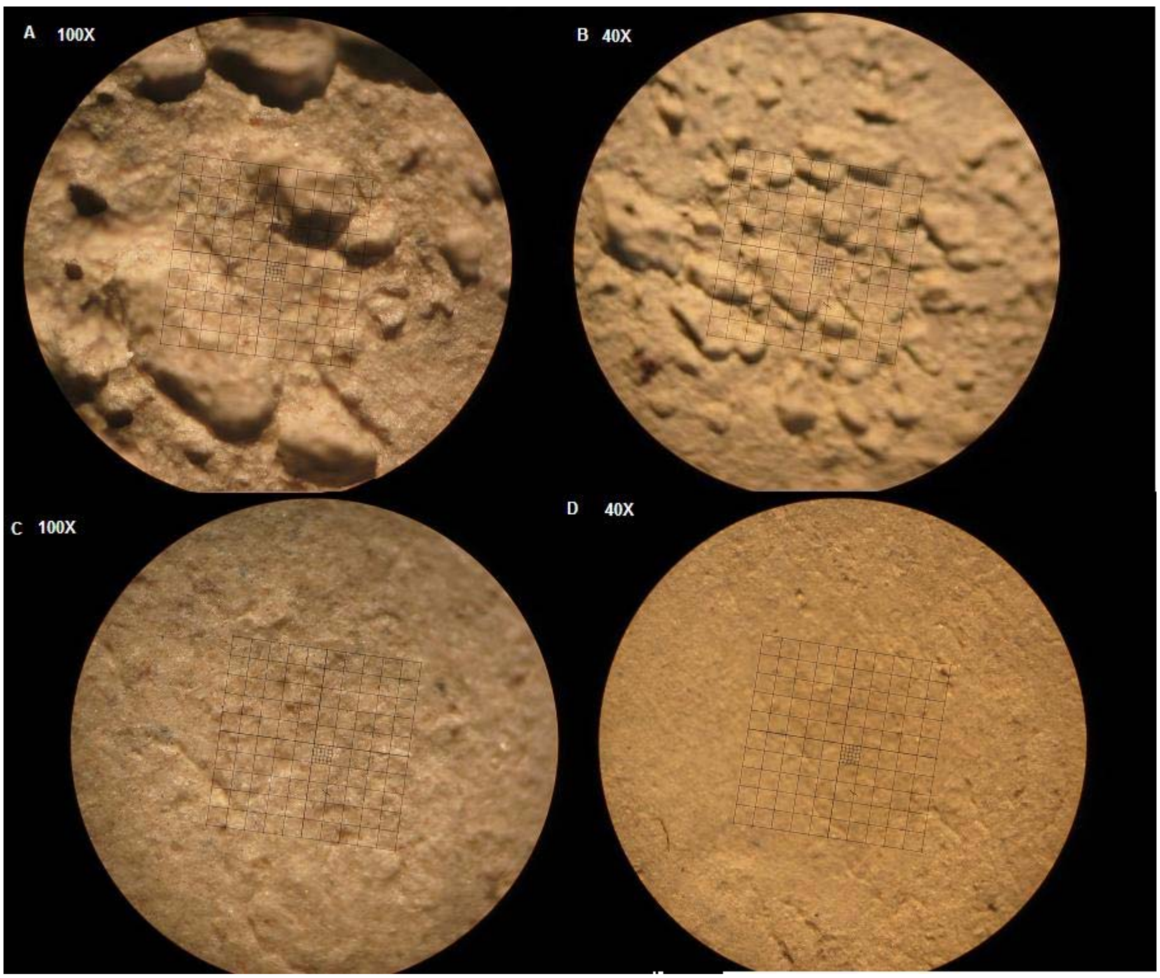

Figure 3 Clay samples as rolled. A&B facing roller, C&D Facing bench

Figure 3 shows that the upside of the disc is much coarser than the downside. Up and down refer to the position of the clay as it is rolled. The down, or bench surface, is covered with a piece of Saran wrap. A large (2.5x7x0.04 in) plastic frame is placed on that. The clay is placed in the frame and manually worked towards the frame. A Saran film is placed on top of the clay. A stiff piece of polypropylene (~50 mil thick) is placed on top of the Saran and the assembly is rolled from above manually. The rolling tends to push the Saran film into the clay causing it to crease. Rolling and working the clay also tends to extract air bubbles from the clay. These two factors cause the upper surface to be rough. The lower surface tends to be pushed flat into the lower saran film. It is expected that a somewhat thicker saran (or other plastic) will inhibit the creasing we currently find.

Table 2 Sample % of original weight after heat treatment

Temperature	Weight	Std. Dev.
66 C	77.7%	0.8%
260 C	77.3%	0.8%
1200 C	70.3%	0.7%

The discs were placed on a woven screen as shown in Figure 6 to promote drying from both surfaces. If the moisture cannot escape uniformly from both sides the disc warps. To improve the flatness during the initial drying we fabricated a plaster slab. Most slip casting molds are made of plaster. The plaster absorbs the water to make the ceramic greenware. The discs are weighted as well to ensure they remain flat.

Once they are finished with the 500 deg F drying, they are shaped to fit the permeation fixture and sanded flat. Then they are placed in the tube furnace for sintering at 1200C.

5.1.2.2 Filtering

Many of the initial samples contained large voids or large particles imbedded in the sanded surface, see Figure 4. After curing the clay shrinks away from the inclusions creating voids. These samples cannot be used for glass deposition. To remove the inclusions, we made a slip with about 300ml clay and 700 ml water. The slip was blended in an Oster blender and filtered. The filtrate amounted to ~5 ml of sand. The supernatant liquid (water) was separated from the clay via sedimentation. This process took several days of air drying to obtain a mixture solid enough to work with.

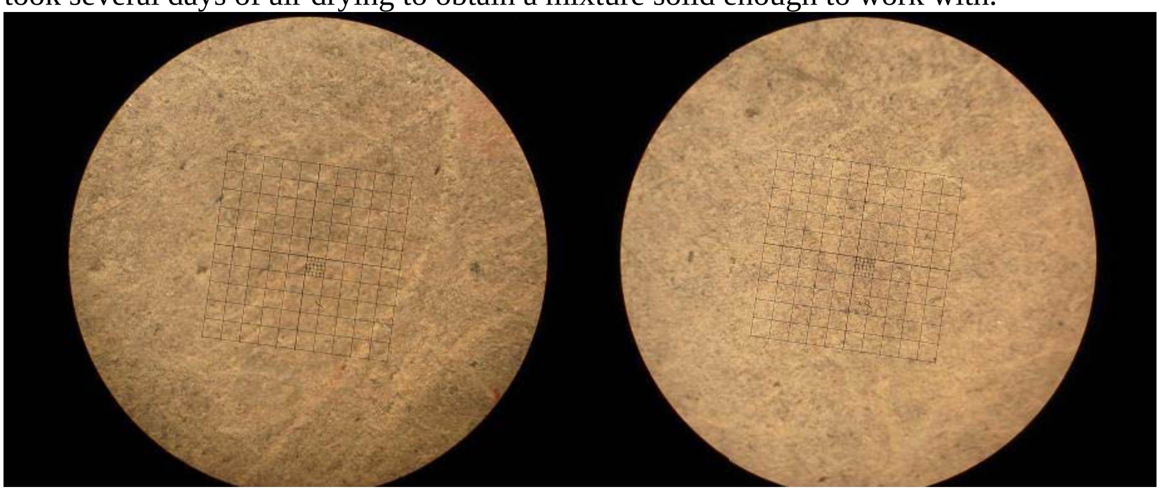

Figure 4 Clay Starch Discs after sanding

Figure 4 shows the discs after firing and sanding. While the final finish is sanded smooth, the grooves can be seen – along with inclusions.

5.1.2.3 Sintering

The dried, sanded discs are fired in a tube furnace at a ramp rate of 3 degrees/minute to 1200C and held at temperature for an hour. The process takes 7.5 hours not including cool down. The cool down rate is not controlled. Any discoloration in the clay from charred organics disappears from the discs during sintering. However, the two discs on the right end of the tube furnace fixture are lighter in color than the other discs, indicating that the temperature in the furnace is not completely uniform, see Figure 5 .

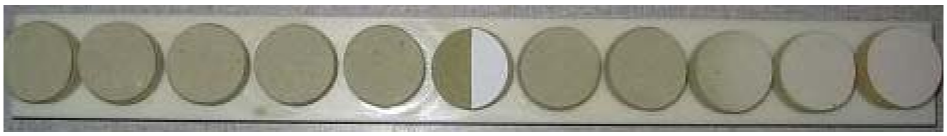

Figure 5 Clay rice starch discs indicate non-uniform heating in the tube furnace

This is most likely due to the insulation used on the right end of the furnace. The processing tube extends beyond the heating element and protrudes approx. 3-4 inches outside of the tube furnace. Although the tube is wrapped in fiberfrax there is still a differential temperature causing heat loss. The left end of the furnace is plugged with firebrick insulation which also supports the tube inside the furnace and prevents it from touching the heating elements. The oven furniture is placed well inside the heating zone in the furnace but the right 2 inches are no longer used for sintering

5.1.2.4 Additives

5.1.2.5 Frit paste

Because the 100% clay fired sample discs are dense, some are gas impermeable. Several experiments were performed using additives in the clay. The first of these experiments used frit paste which is very small particle glass (-325 mesh). The glass flowed during the sintering process and deposited onto the oven furniture. Test results indicated a good flow rate up to 225 sccm at 5 psi differential. However, testing the surface with soap bubble solution showed the flow was permeating through several large holes rather than uniformly over the surface.

5.1.2.6 Rice Starch

An experiment was performed where a 2.5% by weight rice starch/clay mixture was prepared in order to improve the fired porosity of the discs. We based the weight on the 260C dried weight. Hence a 99.3 gm sample of wet clay was mixed with 1.73 gm rice starch.

The dried discs are noticeably darker than the straight clay discs. It is assumed that the rice starch forms a char when the mixture is heat treated at 500F. Figure 6 shows the darkening of the discs. It is also evident that the non-uniform color indicates that the rice starch was not uniformly mixed.

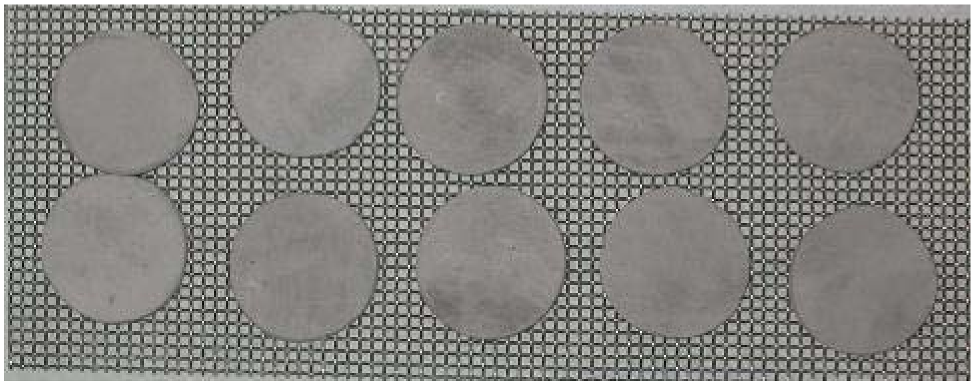

Figure 6 Heat treated clay, rice starch discs

Typically, the discs are sanded smooth before testing. Since these samples were made without filtering the clay of large particles, one of discs was left unsanded for testing. This disc was fairly thick but showed a reasonable permeability with flows to 25 sccm at psi differential pressure. The flow rate through this sample exceeds the flow rates measured through the commercially available alumina discs from Ceramatec. The disc was tested with soap solution afterwards and showed no evidence of a large hole or seal leak. All of the other samples that were sanded had large holes from inclusions. The unsanded surface, however, would be difficult for glass deposition.

A second batch of discs was made using the filtered clay to prevent large holes. The quantity of rice starch was increased to 5% by weight. To improve mixing, the clay was broken up into small bits and then the rice starch was worked into the mixture. This way the mixing was more uniform. Unfortunately, the 5% starch samples were mostly dense. The sanded samples showed better surface properties but two samples were tested and the highest flow rate was 1.4 sccm at 5 psi differential pressure. The discs also showed more pronounced warping. Presumably, the large grains provide some structural stability.

5.1.2.7 Increasing permeability

To increase permeability and obtain improved uniformity of pores, a much larger percentage of starch additive is required. To do this a grog was prepared. The grog is a specially prepared mixture that is fired and then pulverized into a powder. It is then mixed with additional clay and starch and dry pressed after which it is sintered again. The recipe was obtained from a paper on fabricating a low cost water purifier using clays (Reference 1). The paper identifies the pore size to be around 1 micron for kaolin clay and 3-6 micron for red clay. This is the right ballpark for our needs.

To prepare the grog the recipe calls for up to 50% starch to clay ratio. We used 20% starch based on the dry clay weight or 46 grams moist clay (32 gms dry) and 6.5 grams rice starch. The clay was broken up into small bits and the starch was kneaded in. The grog was rolled out into the form using the previous forming techniques. Long strips were cut to be placed in the tube furnace.

Sintering a grog mixture requires a slower ramp time to 500C. We used a rate of 2 degrees/minute. The burning rice starch makes some smoke so the furnace is vented periodically simply by removing the insulation at one end to allow smoke to dissipate. Increasing the temperature too quickly to 500C causes the clay to bloat, blister and crumble and can lead to poor sintering. After rice is burned off, the furnace temp can increase at normal ramp rate to 1200C.

The fired grog was broken up and pulverized using a mortar and pestle. Next, the grog was mixed with more clay and starch using the ratio of 50 parts grog, 40 parts clay & 10 parts starch. Again, the clay contained 30% water and this was factored into the recipe. The resulting mixture was so dry that it could not be worked like regular clay. Two grams of additional water was added to make the mixture more plastic. However, this caused the discs to crack and distort in the drying oven and a new recipe was mixed.

The final composition used was 1 gm grog, 1.14 gm clay (moist) and 0.2 gm rice starch. After mixing the dry ingredients were pressed into a 1 inch diameter circular template placed over a saran covered base. Another piece of saran was placed on top and a roller was used to compress the mixture into the form. Additional dry ingredients were packed in and rolled. Next the mixture was compressed in the hydraulic press to 2000 psi gage or 6,552 psi contact pressure. The pressed coupon was inverted onto a monolith to transport it to the tube furnace for sintering.

5.1.2.7.1 Results

The dry clay mixture inhibits bond formation between clay particles tending to increase permeability. Also, grog particles are prefabricated porous agglomerates due to their high starch content during mixing. This also increases permeability. The coupon surface, shown in Figure 7, shows a more discrete particle appearance rather than a smooth surface which is typical of standard clay. Unfortunately, the surface is also marred by flakes and crevices. This is likely due to insufficient compacting of the dry compound into the mold. We may be able to pack more material in and compress to a higher contact pressure to improve the surface.

Figure 7 Dry pressed clay disc prepared with grog & rice starch

5.1.2.8 Uncoated clay permeation test results

Figure 8 shows the permeance for uncoated clay sample S/N 0033 (lower line). As a reference, the upper line shows the measured permeance through a Cercanam C-16 layer. The clay sample was not optimized but the permeance is in the same range.

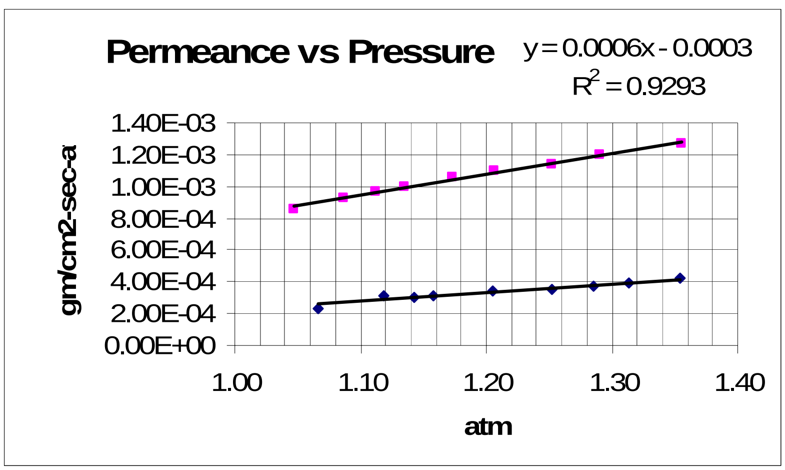

Figure 8 S/N 0033 uncoated clay (lower line) and S/N 0022 Cercanam C-16

5.1.3 CERCANAM samples

We obtained six C-16 CERCANAM samples from Ceramatec. The discs were pre-sintered this time in order to prevent surface disruption and/or dust formation during glass deposition. They will receive a glass coating from Plasma Dynamics using plasma enhanced chemical vapor deposition. Results are not yet available. The permeance is shown in Figure 8 above.

5.2 Glass Coatings

5.2.1 Ion Beam Assisted Deposition (IBAD)

For the option phase, Tufts attempted to deposit coatings using silicon oxide rather than just silicon or added oxygen by way of an ion gun. In previous work, the ion gun often did not deposit at a fast enough rate. The result was that the sample would overheat. Therefore, the deposition rate was reduced to allow heat transfer by radiation to take place. The amount of oxygen in the deposit could not be quantified. By changing to silicon oxide, the composition was known and the deposition rate was markedly increased.

The Ceramatec CERCANAM C-16 sample was unsintered and was polished using a ½ micron polishing paste on the advice of Ceramatec. A 2 micron thick coating was applied. The color was clear rather than the slightly gold to beige colors of previous samples. This was a result of the oxygen. The sample was not annealed but placed in the permeation fixture. Although initial results looked good, the sample cracked during the test as evidenced by a small noise followed by an increased flow rate to 22 sccm at only 1.3 psi differential pressure. The sample was rechecked and resealed. The flow rate remained high. Soap solution showed that the leak was not from the perimeter seal.

The cracked sample may be a result of the surface finish or the applied layer. The polishing may have created a surface with no tooth to it preventing the glass from depositing within the pores. A very slight differential pressure caused the layer to crack or delaminate. The pressure is applied to the glass side which presses into the ceramic so the glass could not be lifted from the surface. However, if the surface had a large pore with a thin layer of glass covering it, the pressure could cause a crack. None of the uncoated samples appeared to have large pores in the surface. All of the IBAD layers had large surface bumps after deposition which never appeared on the baseline glass slide used for thickness measurement. The conclusion was that the un-sintered ceramic surface was compromised somehow during the deposition process. The clay literature advises that it is important to fire the substrate prior to glaze application.

Tufts University services were not available in May or June and had limited availability in April.

5.2.2 EPD

This section describes experimental development work on electrophoretic deposition of glass onto aluminosilicate (clay) discs.

5.2.2.1 Disk 1 Forming

The clay disc used in the experiment is described above and consisted of a 2.5% rice starch doped disc. Microscopic exam showed inclusions in the surface with some empty and some containing large sand grains.

5.2.2.2 Solution

The solution consisted of 0.528 gm Cab-O-Sil EH-5 fumed silica, 100 ml of 94-96% ethanol solvent. The solution was mixed ~2 minutes in a Drinkmaster (high shear mixer). Since the solution was found to have an immeasurably high resistance, 0.2108 gm NaOH (anhydrous) was added. The resistance dropped to 13.8 kOhm as the NaOH dissolved. This rose slightly until the end of the experiment to 20.72 kOhm.

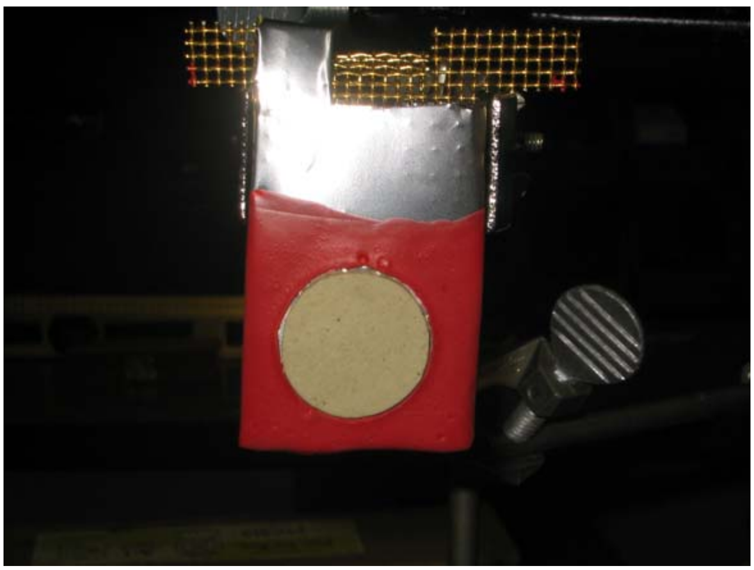

Figure 9 Cathode Electrode

5.2.2.3 Test Rig

The test rig consists of an anode, cathode, beaker, a power supply and two multimeters. The cathode, shown in Figure 9, consists of a 321 stainless steel foil wrapped on a gold plated stainless steel screen. The test disk was glued in place with cyanoacrylate glue. The glue was selected because it easily "unbonds" at temperatures above 150F. The entire cathode was dipped in "Plasti Dip". The area above the disk was cut away.

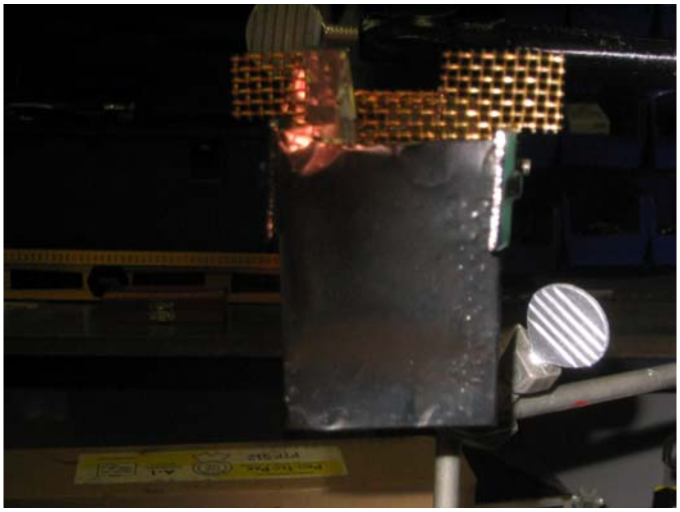

Figure 10 Anode is SS foil wrapped around Au plated, SS screen

The anode is shown in Figure 10 and is made similar to the cathode except there is no plastic insulation or disc. The two electrodes are mounted in an 80 ml beaker. The distance between the two electrodes is about 5/8" (1.6 cm). About 90 ml of solution are required to cover the disk. A Teflon stir bar is dropped into the beaker and the solution is kept mixed during the entire experiment. It was found that the Vinyl coating was damaged by the alcohol and the entire test rig was greatly simplified.

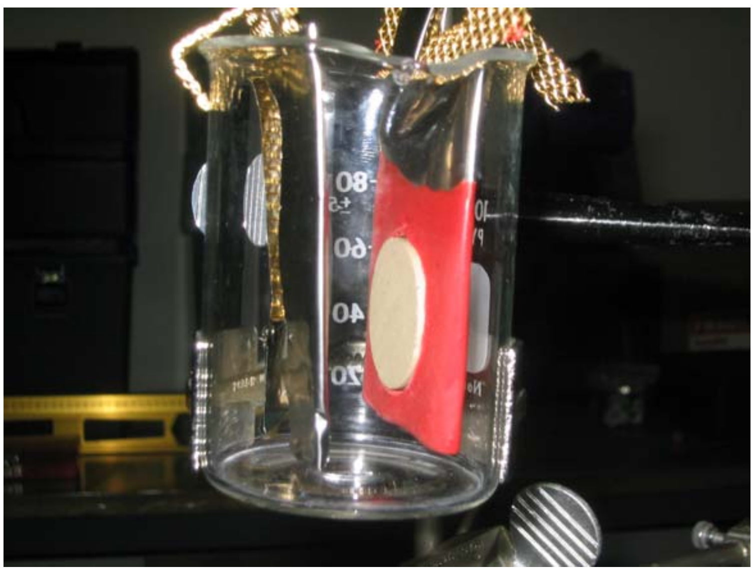

Figure 11 EPD electrodes assembly

5.2.2.4 Procedure

1. Mount the disk on the cathode and insert the electrodes as shown in Figure 11
2. Insert the TFE magnetic stir bar
3. Pour the solution into the beaker until the test disk is immersed and start stirring.
4. The electrodes should be connected to the power supply as shown in Figure 12.
5. With a multi-meter connected across the cell, measure the resistance.
6. When the electrodes are connected, turn on the power supply and raise the voltage to about 10 volts and record the current. The experiment is run potentiostatically.

7. The applied potential is increased to 40 volts in increments of 10 volts and the time and current is recorded.
8. Remove sample from solution
9. Dry sample in 150F oven for 1 hour.
10. Examine microscopically

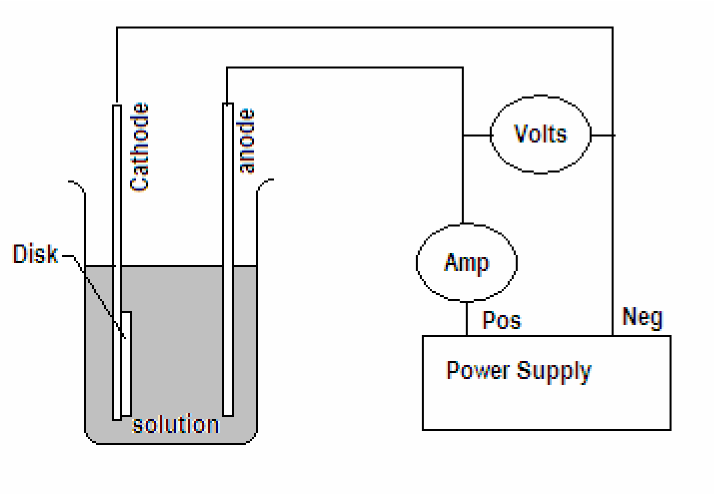

Figure 12 Experiment Schematic Diagram

Figure 13 Results of EPD operations on first sample

5.2.2.5 Results

The results of the electrophoretic deposition of glass on disc 1 are shown in Table 3. The nature of the deposit is affected by the solvent, particle size, applied potential and the distance between the electrodes. In this test the distance between the electrodes was about 1.6 cm. The area of the electrode is 15.6 cm^2 (1.5x1.625 in) for the anode. The cathode was insulated except for the ceramic disk – which is itself an insulator. This means that the current flux varied between 0.72 milliamp/cm2 and 13.14 milliamp/cm2 during the course of the testing. Figure 13 shows the disc after the EPD processing. The disk was dried and then photographed. The figure shows that the glass deposits were leaf like. It indicated that the glass from solution tended to deposit on the highest energy surface. It is also apparent that the deposit is non-uniform. As mentioned, during the test, the cathode plastic coating was damaged.

Table 3 Potentiostatic record of first disk

Time	Volts	Current (ma)	Current Density ma/cm^2
14:38	10	11.3	0.72
14:40	20	33.6	2.15
14:41	28.89	56	3.59
14:42	39.69	85.5	5.48
14:43	"	91.8	5.89
14:44	"	103.4	6.63
15:06	"	175	11.22
15:26		205	13.14

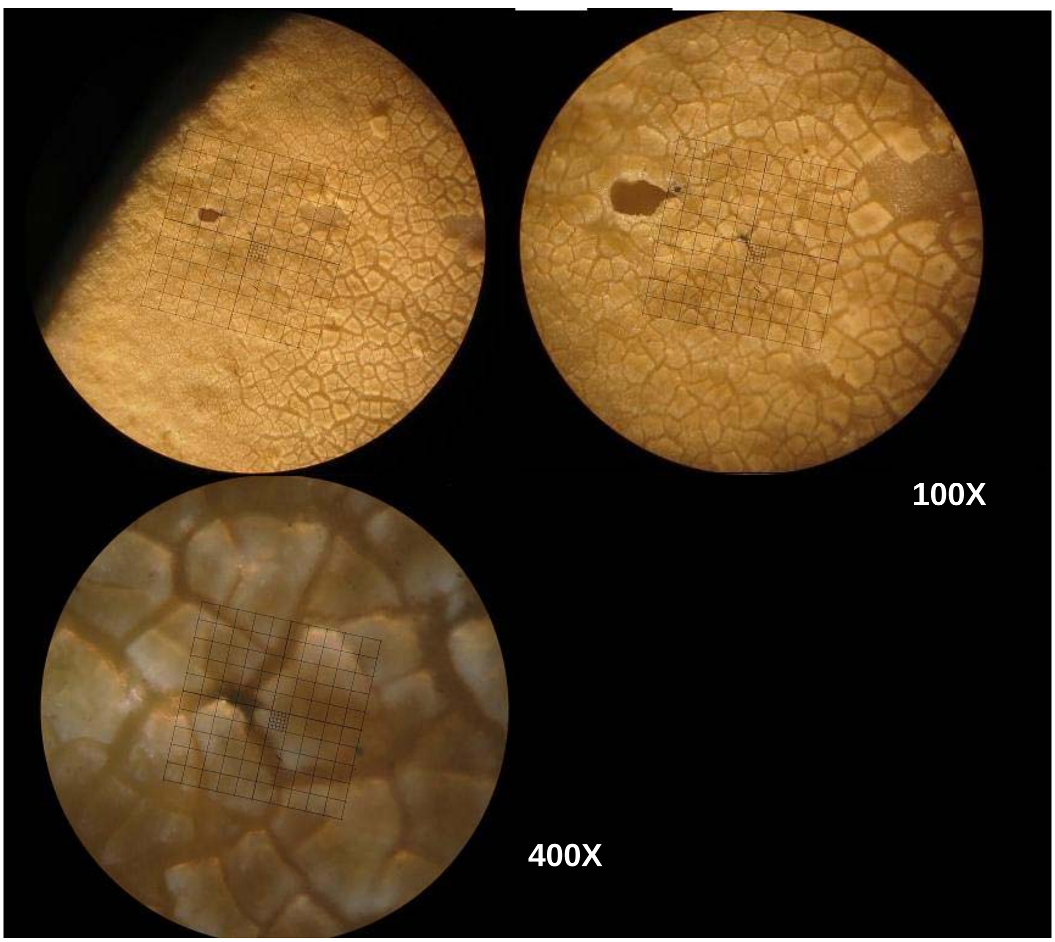

Figure 14 Disc 2 showing mudcracked part of surface

5.2.2.6 *Disc 2*

This disc was identical to disc 1. The test rig was greatly simplified and the electrode areas were reduced to approximately the size of the disk or 1 in diameter. This yields an area of 0.785 in2 (5.02 cm2) or about 3 times smaller than the disk 1 test. The electrodes were slightly closer together, ~0.5 in. The solution used was the left over solution from the disc 1 test. The results were quite different. The deposit formed had a "mudcracked" surface in a small region of the disc. Presumably it was in an area where the two electrodes were closer together. Surprisingly, the stainless steel foil anode was coated with a similar deposit.

Table 4 Disc 2 Operating conditions

Time	Volts	Current (ma)	Current Density ma/cm^2
13:47	14.48	33.28	6.29
13.52	38.07	97.9	19.50
13:57	38.06	104.5	20.82
15:00	38.11	76.7	15.28
16:52	38.15	53.3	10.62

After sintering the mudcracked surface material appears to have formed balls of glass on the surface. This indicates that the clay surface is non-wetting. Commercial glazes use metal oxides as fluxes to improve adhesion. Microwaving the sample did not appear to affect the surface.

5.3 Glaze

Commercial liquid glaze by American Art Clay Co. was applied to several substrates using an air brush. The air brush is superior to a paint brush in that it applies a thin uniform coating without leaving brush strokes on the surface. We used a fritted colored glaze. Processing the glaze in the microwave was unsuccessful. The microwave did not reach the proper firing temperature and only half of the sample glaze melted. The other half appeared unchanged. The resulting stress on the layer caused it to crack. This triggered the purchase of the tube furnace.

Several glazed samples were successfully prepared in the tube furnace which can reach 1200C. One application of glaze was applied to pre-sintered clay discs. The glaze was then sintered to 1200C. Serial number 0035 showed a fully dense coating. The identical recipe uncoated disc was permeable. Thus, we successfully obtained a fully dense glass coated porous ceramic.

5.4 High temperature permeation apparatus

The high temp setup, shown in Figure 15, consists of a 1 inch diameter glass outer tube with a 3/8 inch diameter glass inner tube. The glass is vycor which is good to a continuous use temperature of 900C. We used standard brass and stainless tube fittings with an added o-ring seal behind the cone shaped ferrule to seal to the glass tubes. The 3/8" port on the tee fitting was bored out to allow the 3/8" tube to pass through it into the elbow. The apparatus was setup outside the tube furnace in order to show the configuration. The glass is placed inside the furnace with the element in the vertical position.

Figure 15 High temperature test apparatus shown without tube furnace

6 CONCLUSION

6.1 Ceramic Intermediate Layer

Our work to date has shown that we are able to achieve permeable kaolin clay substrates suitable for glass deposition. The clay is mixed with an additive to achieve the desired permeability since solid clay compositions are dense. The key to achieving a uniformly porous clay sample is to limit inter particle bonding and to spike the formula with porous clay agglomerates, grog. The grog serves two purposes, one being a prefabricated porous structure of the same material and two adding structural strength to the mixture to inhibit warping. Although the grog laced samples were not fully optimized, it is apparent from the photomicrographs that the grog limits interparticle bonding as evidenced by the grainy texture. The dry pressing operation produces a thin disk that doesn't warp since there is no shrinkage due to water loss. Thus the pore structure and porosity of the presintered sample is likely similar to the post sintered sample. The measured permeance is close in range to the commercially available alumina made by Ceramatec.

Slip casting clay must be carefully controlled. Wall thickness and moisture loss are key factors. Plaster molds are a convenient method for absorbing moisture from the sample during cure. Thin, flat shapes are very difficult to achieve, therefore, clay slip casting was not pursued. However, a plaster slab was successfully employed to limit warping during drying of the rolled clay.

Slip casting of the Cotronics 940LE was unsuccessful as well. This was mainly due to the chemical reaction between powder and activator which generates bubbles. Adjusting viscosity by modifying the quantity of liquid and order of mixing were not reliable. Attempts to control particle interaction by using acid caused additional side effects. This method was abandoned.

6.2 Glass Layer

Ion beam assisted deposition (IBAD) of silicon oxide was unsuccessful and became unavailable in May. All tested IBAD samples showed an unacceptable level of permeance in the room temperature apparatus. Coatings were applied to cast 940LE coupons, CERCANAM coupons and porous tubes. Plasma targets included pure silicon, silicon with added oxygen and silicon dioxide. Coating thickness ranged from just under 1 micron to 2.2 microns. Coatings were tested as received, annealed and multiple thermal cycled. Post inspection of the layers showed large mounds of material on the surface which were not present prior to coating. Causes may include not sintering the material prior to deposition and contamination of the plasma with dust and debris from either the apparatus or the coupon itself.

In contrast, glazes were applied successfully to the clay surface. A fully dense coating was achieved. The fritted Amaco glaze was applied using an air brush. Only one layer was sufficient to obtain a pore free surface. We have not as yet determined if the glass is permeable to hydrogen. The high temperature apparatus is being completed and will undergo shakedown testing shortly.

Electrophoretic deposition (EPD) was also employed as an alternative glass application method. Cab-O-Sil fumed silica was suspended in a solvent and deposited onto the coupon surface using a power supply. The solvent was optimized to improve conductivity and the test fixture was optimized as well. The surface energy of the coupon is important in the resulting layer morphology. A microscopically rough surface will produce a rough EPD coating. Another important factor is the wetting properties of the coupon. Glazes contain metal oxide compounds as fluxes. Post heating of the EPD coating indicates a flux may be needed to produce a smooth coating.

The glaze and frit pastes added to the clay flowed during sintering which is advantageous for depositing into pores. The soak or dwell time of the furnace may be important to prevent too much run off and/or obtain a thin, contiguous coating.

References

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